

	Type	L #	Hits	Search Text	DBs
1	BRS	L1	75298	microwave or micro adj wave	USPAT
2	BRS	L2	493	1 and 422/50-138.ccls.	USPAT
3	BRS	L3	415	1 and 436/1-183.ccls.	USPAT
4	BRS	L4	696	2 3	USPAT
5	BRS	L5	330	lautenschlager.in.	USPAT
6	BRS	L6	19	1 and 5	USPAT
7	BRS	L9	2830	1 near4 (vessel or container or bomb or reactor)	USPAT
8	BRS	L10	2726	9 not 4 not 5	USPAT
9	BRS	L11	360	10 same (digest\$ or reaction or combinatorial)	USPAT
10	BRS	L12	90625	microwave or micro adj wave	JPO; DERWENT
11	BRS	L13	3638	12 near4 (vessel or container or bomb or reactor)	JPO; DERWENT
12	BRS	L14	364	13 and (digest\$ or reaction or combinatorial)	JPO; DERWENT

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(FILE 'HOME' ENTERED AT 10:34:24 ON 21 JUL 2005)

FILE 'CA' ENTERED AT 10:34:32 ON 21 JUL 2005

L1 96434 S MICROWAVE OR MICRO WAVE
L2 2096 S L1(5A) (VESSEL OR CONTAINER OR BOMB OR REACTOR)
L3 7190 S L1(8A) (DIGEST? OR REACTION OR COMBINATORIAL)
L4 680 S L2 AND L3
L5 442 S L2 AND PRESSURE
L6 184 S L4 AND L5
L7 355 S L2(8A) (CAP OR CLOSED OR SEALED OR SEAL OR LID)
L8 111 S L7 NOT L3
L9 41 S L8 AND (EXTRACT? OR REACTOR OR FLEXIBLE OR SYNTH?)
L10 225 S L6,L9
L11 188 S L10 NOT PY>2002
L12 0 S L11 NOT L10 AND PATENT/DT AND PY<2004

=> d 111 1-188 bib,ab

L11 ANSWER 20 OF 188 CA COPYRIGHT 2005 ACS on STN
AN 135:368793 CA
TI Lead determination in ultra micro samples of animal diet using graphite furnace atomic absorption spectrometry
AU Iavicoli, Ivo; Castellino, Nicolo; Carelli, Giovanni; Schlemmer, Gerhard
CS Institute of Occupational Health, School of Medicine, Universita Cattolica del Sacro Cuore Largo Francesco Vito, Rome, 1-00168, Italy
SO Atomic Spectroscopy (2001), 22(3), 319-323
AB A graphite furnace at. absorption spectrometry method was developed for the detn. of very low levels of Pb in small samples of animal diet. The method is based on a **microwave**-assisted **pressure vessel digestion** using miniaturized vessels for sample masses of 100 mg. Due to the small acid vol., the diln. factor during digestion was only 10. Detection limits of 5 µg/kg Pb were achieved in a complex matrix consisting of mainly caseine 22% (wt./wt.), corn starch 28.6%, sucrose 30%, cellulose 4%, soya oil 7%, minerals 5.2%, vitamins 2%, and DL methionine 0.4%. The reproducibility of the method was 15% at a Pb concn. of 37 µg/kg and 11%, resp., using the std. addns. method before and after digestion. The recoveries of Pb added to the samples prior to or after digestion were 83.2 ± 3.0% and 82.3 ± 2.2%, resp.

L11 ANSWER 24 OF 188 CA COPYRIGHT 2005 ACS on STN
AN 134:368666 CA
TI Parallel synthesis in the microwave field
AU Nuchter, Matthias; Lautenschlager, Werner; Ondruschka, Bernd; Tied, Antje
CS Institut fur Technische Chemie und Umweltchemie (ITUC), Institut fur Technische Chemie und Umweltchemie (ITUC), Friedrich-Schiller-Universitat Jena, Jena, D-07743, Germany
SO LaborPraxis (2001), 25(1), 28-31
LA German
AB Two **microwave** app. for parallel **combinatorial** syntheses are presented. In the 1st one, a rotor receives 36 reaction vessels of 50 mL useful vol. each arranged in 2 concentric rows. The vessels are fitted with

individually adjustable magnetic stirrers and their sealing caps serve as **pressure** release valves adjustable between 5 and 20 bar. The temp. is monitored by fiber optical sensors and contactless IR measurement. Parallel reactions conducted under identical conditions indicate a homogeneous energy distribution in the microwave field. Another app. receives some deep-well plates for 96 samples each, which are moved around a horizontal axis. Glass vessels of 2 mL vol. are inserted into the wells and the plates are **pressure**-sealed.

L11 ANSWER 34 OF 188 CA COPYRIGHT 2005 ACS on STN

AN 133:334546 CA

TI Digestion of NIST peach leaves using sealed **vessels** and inexpensive **microwave** ovens

AU Bell, Paul F.; Xie, Bin; Higby, Jeffery R.; Aminha, Navine

CS Agronomy Dept., Louisiana State Univ. Agric. Center, Baton Rouge, LA, 70803, USA

SO Communications in Soil Science and Plant Analysis (2000), 31(11-14), 1897-1903

AB **Microwave**-oven **digestion** of plant materials for the detn. of plant-nutrient concns. is an aggressive and effective dissoln. procedure. Com. ovens are expensive, however, and ovens designed for home use may provide an inexpensive alternative. Evaluated were three microwave ovens: Panasonic NN5510, Sharp Carousel II, and a com. unit CEM MDS-2000; and two Teflon vessels: Savillex 571R2 and CEM 327011 for their ability to digest batches of six or twelve plant samples and to compare their analyses with those from conventional block-heater digestions. For the **microwave**-oven **digestions**, was placed in each vessel 0.1 g National Institute of Stds. and Technol. (NIST) std. ref. material (SRM) 1547 peach leaves, 1.0 mL conc. HNO₃, and 0.6 mL 30% w/v H₂O₂. The digestates were stirred, soaked for 15 min., heated in a rotating carousel as described below, cooled 15 min, added 8.4 mL deionized-distd. water to make 10 mL vol., filtered, and analyzed with ICP-AES. The block-heater method used a nitric/peroxide digestion, unsealed glass tubes, 0.5 g samples and a 25 mL diln. There were no differences between Ca, K, S, P, Zn, Cu, and Fe concns. of NIST SRM 1547 when samples were **digested** using com. or domestic **microwave** ovens ($P > 0.05$) and recoveries ranged from 84 to 95% of NIST certified values. Recoveries were greater from the block heater **digestions** than with the **microwave digestions** from four of the seven elements analyzed ($P < 0.05$). Variability was greater from the **microwave** oven **digestions** than the block heater. Home-use **microwave** ovens used for the **digestion** of plant materials are to be set to 300 to 350 W and heated for ≥ 30 min. for six-vessel digestions and 45 min. for twelve-vessel batches. The procedure produces low vessel **pressures** (≥ 410 kPa) with little chance of relief valves rupturing and the loss of samples. The domestic ovens may be useful for those requiring occasional analyses or those who analyze samples of similar nature negating the need for frequent method modification. Lab. personnel must adhere to the recommended sample sizes and reagent vols. to prevent explosions.

L11 ANSWER 41 OF 188 CA COPYRIGHT 2005 ACS on STN

AN 133:12119 CA

TI Rapid sample preparation using closed-vessel **microwave digestion** for

determining trace metals in fish tissue and sediment

AU Van Staden, J. F.; Van der Merwe, L.; Kempster, P. L.; Van Vliet, H. R.
 CS Department of Chemistry, University of Pretoria, Pretoria, 0002, S. Afr.
 SO South African Journal of Chemistry (2000), 53(1), 23-27

AB Two **microwave digestion** procedures using closed **vessels** with **pressure** relief septa were developed for sample prepn. of fish tissue and sediment, prior to emission spectroscopic anal. for trace metals. Nitric and perchloric acids were used for the fish tissue digestion; and nitric, hydrochloric and hydrofluoric acids for the sediment digestion. The performance of the digestion procedures were evaluated relative to open beaker digestion using international ref. material, as well as real samples. Analyses were conducted with an Inductively Coupled Plasma (ICP) Emission Spectrometer. The closed vessel digestions showed more complete digestion and less contamination from the air than open beaker digestion.

L11 ANSWER 44 OF 188 CA COPYRIGHT 2005 ACS on STN
 AN 132:250161 CA
 TI A new generation of high-**pressure microwave** sample **digestion** systems
 AU Baasner, J.; Schulze, H.; Kainrath, P.; Kettisch, P.
 CS USA
 SO Spectroscopy (Eugene, Oregon) (1999), 14(6), 42-44
 AB The capabilities of a closed-**vessel**, high-temp., high **pressure microwave digestion** system for both org. and inorg. matrixes are outlined. Data are reported showing the system's capabilities in reducing the potential for matrix-related interferences in AA, ICP-OES, ICP-MS, and electrochem. techniques in the anal. of food and other samples.

L11 ANSWER 50 OF 188 CA COPYRIGHT 2005 ACS on STN
 AN 131:273479 CA
 TI **Flexible** vessel and frame for microwave assisted chemistry
 IN Hargett, Wyatt Price, Jr.; Thomas, James Edward; Barrett, Matthew Donald
 PA Cem Corporation, USA
 SO PCT Int. Appl., 21 pp.
 PI WO 9954034 A1 19991028 WO 1999-US8728 19990420
 US 6136276 A 20001024 US 2000-482453 20000113
 PRAI US 1998-62858 A1 19980420

AB A self venting sealable vessel system for microwave assisted chem. is disclosed. The system includes a vessel formed of a microwave-transparent material, one end of which forms an opening for placing materials inside the vessel, a lid for being seated against the opening, a **flexible** frame surrounding the vessel and lid and formed of a microwave-transparent material, and means for urging the frame against the vessel and seated lid with a predetd. force to seal the vessel at low pressures and so that the frame refrains from flexing until the pressure inside the vessel exceeds the predetd. force, after which the frame flexes sufficiently to allow the lid to unseat and gases to vent safely from the vessel without rupturing the vessel or the frame.

L11 ANSWER 54 OF 188 CA COPYRIGHT 2005 ACS on STN
 AN 131:211119 CA
 TI Development of a High-**Pressure** Asher Focused Microwave System for Sample Preparation

AU Matusiewicz, Henryk
 CS Department of Analytical Chemistry, Politechnika Poznanska, Poznan, 60-965, Pol.
 SO Analytical Chemistry (1999), 71(15), 3145-3149
 AB The development of high-**pressure** Asher focused **microwaves** (HPA-FMs), a novel approach to **microwave** assisted **digestion**, is described. The system uses focused **microwaves**, at 2.45 GHz, to improve **digestion** capability with up to 650 W **microwave** power concd. into six quartz **pressure** vessels contg. samples and nitric acid. The device combines **microwave** heating with high-**pressure vessel** technol. (**reactions** can be conducted at **pressures** and temps. up to 130 bar and 320°, resp.). Methodol. was developed using powd. biol. ref. material. The residual carbon content of digested bovine liver sample was detd. by Coulometry after combustion in an oxygen stream to evaluate the effectiveness of the decompn. procedure. With this new decompn. device, org. material is totally oxidized with nitric acid in a single-step digestion.

L11 ANSWER 76 OF 188 CA COPYRIGHT 2005 ACS on STN
 AN 128:330421 CA
 TI Closed **vessel microwave**-assisted wet **digestion** with simultaneous control of **pressure** and temperature in all vessels
 AU Zischka, Michael; Kettisch, Peter; Schalk, Andreas; Knapp, Guenter
 CS Institute Analytical Chemistry Micro- Radiochemistry, Technical University Graz, Graz, A-8010, Austria
 SO Fresenius' Journal of Analytical Chemistry (1998), 361(2), 90-95
 AB A novel system for **microwave**-assisted wet **digestion** in closed **vessels** is described. Six **pressure** vessels made of quartz or Hostaflon TFM are placed in a special rotor in a microwave oven. During sample decompn. the **pressure** and temp. are measured in each vessel, and the data are transferred from the rotor by IR light to the control unit. This means that no pneumatic or electronic connections to the vessels need to be established, providing very easy handling. The temp. history of each vessel is recorded; so the progress of each sample digestion may be reconstructed later on. Thus the requirements for quality control in sample digestion are available for the first time. For sample digestion at 75 bar and $\leq 280^\circ$, quartz vessels with 50 mL vol. are used. Depending on the matrix, the max. sample loading capacity is 1.2 g. TFM-vessels with 100 mL vol. work at 30 bar $\leq 240^\circ$. The performance of the digestion system was tested with 12 std. ref. materials. The results closely matched the certified values.

L11 ANSWER 83 OF 188 CA COPYRIGHT 2005 ACS on STN
 AN 128:42984 CA
 TI Sample decomposition-Quo vadis
 AU Knapp, Gunter
 CS Department Analytical Chemistry Micro- and Radiochemistry, Technical University Graz, Graz, Austria
 SO Contemporary State and Trends of Decomposition Methods in Analytical Chemistry, Proceedings, Slovak-Austrian Symposium, Kosice, Slovakia, Feb., 1997 (1997), 10-17. Editor(s): Krakovska, Erika. Publisher: Stroffek Publishing, Kosice, Slovakia.
 AB A review, with 12 refs., is given on sample prepn. techniques particularly decompn. Sample decompn. is the most significant sample

prepn. technique for trace element anal. Important functions of sample digestion are dissoln. of solid materials and oxidn. of org. matrix constituents. The detn. of elements in the trace and ultratrace range make great demands on decompn. methods. With respect to correct anal. results the classical decompn. techniques fusion, oxidn. with oxygen and wet digestion were improved to reduce systematic errors caused by contamination or losses of elements. In anal. chem. quality control becomes more significant increasingly. But presently it is applied however, 1st of all to measuring techniques and not to sample prepn. One of the most important sample prepn. methods in element anal. is **microwave** assisted wet **digestion** in closed pressurized vessels. The parameters important for quality control are in this case **pressure** and temp. **Pressure** control is important because the closed vessels are tight only up to a certain **pressure**. During the decompn. procedure this **pressure** may be exceeded under no circumstances. The temp., on the contrary, is responsible for the efficiency of the decompn. reaction. The higher the temp., the higher is the oxidn. potential of the digestion reagent. Recording the time temp. curve of the decompn. process the extent of sample degrdn. can be established afterwards. New developments enable recording of **pressure** and temp. of all **vessels** in a **microwave** oven during **digestion**. **Microwave** assisted flow **digestion** systems open up new possibilities in fully automated sample prepn. for element anal. For fast and almost complete sample digestion the temp. must be as high as possible. For this reason elevated **pressure** has to be applied within the flow digestion systems. For an extensive oxidn. of org. sample constituents with nitric acid temps. of $>220^{\circ}$ are necessary. Teflon tubes used, however, do not withstand the vapor **pressure** of the digestion mixt. at 220° or more. Thus new alternatives has to be found to overcome this limitation.

- L11 ANSWER 86 OF 188 CA COPYRIGHT 2005 ACS on STN
 AN 127:360356 CA
 TI Method and device for contact-free **pressure** measurement in pressurized decomposition **vessel** for thermally and **microwave**-heated **reactions**
 IN Kraemer, Rainer
 PA Berghof Laborprodukte Gmbh, Germany
 SO Ger. Offen., 8 pp.
 PI DE 19710499 A1 19971030 DE 1997-19710499 19970313
 PRAI DE 1997-19710499 A1 19970313
 AB The **pressure** formed inside the vessel is transferred to a sensor strongly linked with the vessel and comprising a transparent, amorphous, **pressure**-induced, and double-refractive material. The **pressure**-induced double refraction of the sensor changes the polarization of the polarized beam irradiating the sensor, which measures the inner **pressure** of the vessel.
- L11 ANSWER 97 OF 188 CA COPYRIGHT 2005 ACS on STN
 AN 126:173438 CA
 TI A novel **microwave** autoclave for automation of high **pressure** chemical **reaction** applications
 AU Lautenschlaeger, W.; Engelhart, W.G.; Metzger, M.; Visinoni, F.
 CS MLS/Milestone s.r.l., SORISOLE, 24010, Italy
 SO Process Technology Proceedings (1996), 12(High Pressure Chemical

Engineering), 693-700

AB To avoid manual operation in operation of industrial microwave-based batch autoclaves, a new microwave autoclave was designed that eliminates manual operation by combining **microwave** heating with high-**pressure vessel** technol. to carry out chem. reactions at up to 200 bars and 350°. The system is specifically designed for semi-automated batch processing of multiple samples. Full automation can be achieved by interfacing the autoclave with a lab. robot arm to load and unload sample racks. An example was given for manuf. of pharmaceuticals.

L11 ANSWER 105 OF 188 CA COPYRIGHT 2005 ACS on STN

AN 125:74836 CA

TI Comparison of acid mixtures in high-**pressure microwave digestion** methods for the determination of the total mercury in sediments by cold-vapor atomic absorption spectrometry

AU Zhou, Chao Yan; Wong, Ming Keong; Koh, Lip Lin; Wee, Yeow Chin

CS Department Chemistry, National University Singapore, Singapore, 119260, Singapore

SO Analytical Sciences (1996), 12(3), 471-476

AB Four closed-**vessel microwave digestions** methods were compared for the accurate detn. of Hg in sediment by flow-injection cold-vapor at. absorption spectrometry. Several acid mixts. (HNO₃/H₂SO₄, HNO₃/HClO₄, HCl/HNO₃ and HCl/HNO₃/HF) completely digested the soil matrix for the detn. of Hg. The method using aqua regia is preferred because it is time saving, less dangerous and suitable for other trace metal analyses. The merits of **pressure-feedback microwave digestion** is that it simplifies soil sample **digestion**, and there is no loss of Hg. The digestion methods were evaluated by detg. Hg in NIST SRM 1645 River Sediment, NIES CRM No. 2 Pond Sediment and NRCC BCSS-1 Marine Sediment. Recoveries of 92-108% were achieved. Good recoveries of 94-104% were also obtained for soil and marine-sediment samples spiked with different species of Hg.

L11 ANSWER 138 OF 188 CA COPYRIGHT 2005 ACS on STN

AN 120:190375 CA

TI Microwave-Assisted **Extraction** of Organic Compounds from Standard Reference Soils and Sediments

AU Lopez-Avila, Viorica; Young, Richard; Beckert, Werner F.

CS Midwest Research Institute, California Operations, Mountain View, CA, 94043, USA

SO Analytical Chemistry (1994), 66(7), 1097-106

AB As part of an ongoing evaluation of new sample prepn. techniques by the U.S. Environmental Protection Agency (EPA), esp. those that minimize waste solvents, microwave-assisted **extn.** (MAE) of org. compds. from solid materials (or "matrixes") was evaluated. Six certified ref. materials contg. polynuclear arom. hydrocarbons (PAHs) and a few base/neutral/acidic compds., all of which are common pollutants of interest to the EPA, were subjected to MAE in a **closed-vessel microwave** system with hexane/acetone (1:1) at different temps. (80, 115, and 145 °C) and for different periods of time (5, 10, and 20 min). For comparison, the same samples were subjected to room-temp. **extn.** by allowing the solvent mixt. to stay in contact with the solid matrix the same amt. of time as the microwave-**extd.** sample (including any cooling

time). Whereas the av. recovery at room temp. was ~52%, the MAE recoveries for the 17 PAHs (3 of which were deuterated PAHs that were spiked into these matrixes) from the six matrixes were 70% at 80 °C, 75% at 115 °C, and 75% at 145 °C. Although the av. recoveries increased slightly with **extn.** time, the increase was not statistically significant. The performance of the technique varied with the matrix and the analyte. Eleven PAHs had av. recoveries in the 65-85% range, and three compds. (acenaphthene, benzo[a]pyrene, and fluorene) had recoveries of ~50%. The spiked-compd. recoveries were 77% for acenaphthene-d10, 105% for fluoranthene-d10, and 85% for benzo[a]anthracene-d12. Expts. with 14 phenols and 20 organochlorine pesticides indicated that MAE is a viable alternative to the conventional Soxhlet/Soxtec and sonication techniques. The MAE technique requires smaller amts. of org. solvents, and sample throughput is increased by shorter **extn.** times (10 min) and by simultaneous **extn.** of up to 12 samples.

L11 ANSWER 147 OF 188 CA COPYRIGHT 2005 ACS on STN
AN 119:130630 CA
TI Ventable rupture diaphragm-protected **container** for heating contained materials by **microwave** radiation
IN Hargett, Wyatt P.; Littau, Sara E.
PA CEM Corp., USA
SO U.S., 16 pp. Cont. of U.S. Ser. No. 404,693, abandoned.
PI US 5230865 A 19930727 US 1992-948134 19920921
PRAI US 1989-404693 B1 19890908
AB A ventable container, for materials to be heated (preferably **digested**) by **microwave** radiation, includes a movable rupture diaphragm in a venting passageway in a closure for such container. Such diaphragm is tightenable into place so that the container is sealed and in such sealed state is protected by the rupture diaphragm against **pressures** that could develop in the container that are higher than that for which it was designed. The rupture diaphragm is controllably movable, when desired, to open positions to vent the container during heating and afterward, and is resealable after such venting. Because of the reactivity of the reagents utilized in digestions, an inner chem. resistant liner is desirably employed in conjunction with a phys. stronger outer body, with the outer body giving the liner enough strength to withstand **pressures** developed during the heating. Also within the invention are processes for heating and **digesting** materials in the described **containers** with **microwave** radiation, **microwave** heating app. including the invented **containers**, and lined containers, where the liners are of improved sealing structures so that the containers are more tightly sealed when heated.

L11 ANSWER 152 OF 188 CA COPYRIGHT 2005 ACS on STN
AN 118:265539 CA
TI Development and application of a **microwave digestion pressure vessel**
AU Pougnet, M. A. Bruno; Schnautz, Norman G.; Walker, Alta M.
CS Dep. Chem., Univ. Cape Town, Rondebosch, 7700, S. Afr.
SO South African Journal of Chemistry (1992), 45(4), 86-9
AB The design of a **microwave pressure digestion vessel** is presented. The wide practical utility of this device is demonstrated through

applications for prepn. of biol. and geol. materials. Digestion conditions and anal. data are given for a variety of samples including several biol. ref. materials, liver, coal, limestone and iron ores.

- L11 ANSWER 153 OF 188 CA COPYRIGHT 2005 ACS on STN
AN 118:8705 CA
TI High-pressure and microwave **reactor**
IN Lautenschlaeger, Werner
PA MLS GmbH, Germany
SO Ger. Offen., 10 pp.
PI DE 4114525 A1 19920820 DE 1991-4114525 19910503
PRAI DE 1991-4105094 A1 19910219
AB The **reactor** includes a housing of ≥ 1 pressure vessel(s) from a high pressure-resistant material with microwave-transmitting and pressure tightly-**closed** openings and an inserted **vessel** of at least partly **microwave**-transmitting sample holder and a high pressure-resistant, microwave-transmitting, opening-closing element. Several versions of the **reactor** are disclosed.
- L11 ANSWER 155 OF 188 CA COPYRIGHT 2005 ACS on STN
AN 117:48867 CA
TI A new **reaction vessel** for accelerated syntheses using **microwave** dielectric super-heating effects
AU Baghurst, David R.; Mingos, Michael P.
CS Inorg. Chem. Lab., Univ. Oxford, Oxford, OX1 3QR, UK
SO Journal of the Chemical Society, Dalton Transactions: Inorganic Chemistry (1972-1999) (1992), (7), 1151-5
AB Using a thick-walled glass reaction vessel a no. of transition metal organometallic and coordination compds. have been synthesized using microwave dielec. super-heating effects at 40-60° maintaining controlled **pressure** at 10 atm. E.g., $[\text{RuL}_2][\text{PF}_6]_2$ (L = 1,4,7-trithiacyclonane) was prepd. from $\text{RuCl}_3 \cdot 3\text{H}_2\text{O}$, L, and MeOH by microwave heating at 500 W in 96% yield.
- L11 ANSWER 180 OF 188 CA COPYRIGHT 2005 ACS on STN
AN 105:130099 CA
TI Microwave energy for acid decomposition at elevated temperatures and **pressures** using biological and botanical samples
AU Kingston, H. M.; Jassie, L. B.
CS Cent. Anal. Chem., Natl. Bur. Stand., Gaithersburg, MD, 20899, USA
SO Analytical Chemistry (1986), 58(12), 2534-41
AB A closed **vessel microwave digestion** system is described. In situ measurement of elevated temps. and **pressures** in closed Teflon PFA vessels during acid decompn. of org. samples is demonstrated. Temp. profiles for the acid decompn. of biol. and botanical std. ref. materials are modeled by the dissolving acid. Microwave power absorption of nitric, hydrofluoric, sulfuric, and hydrochloric acids is compared. An equation is applied to acid microwave interactions to predict the time needed to reach target temps. during sample dissoln. Reaction control techniques and safety precautions are recommended.

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